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Research Report

Final Technical Report: Addendum One / March 30, 1969

DEVELOPMENT OF TECHNIQUES TO IMPROVE BLADDER MATERIALS AND TEST METHODS

Prepared for:

JET PROPULSION LABORATORY
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PASADENA, CALIFORNIA 91103

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Final Technical Report: Addendum One / March 30, 1969 *March 30, 1969*

DEVELOPMENT OF TECHNIQUES TO IMPROVE BLADDER MATERIALS AND TEST METHODS

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CONTRACT NO. 951484
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S R I Project PRD 6068

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FOREWORD

This Final Technical Report: Addendum One summarizes the work performed by Stanford Research Institute under a continuation to March 30, 1969, of Contract No. 951484 for the Jet Propulsion Laboratory.

The Technical Representative for the Jet Propulsion Laboratory was Mr. Howard B. Stanford; the technical effort at Stanford Research Institute was under the direction and guidance of Dr. R. F. Muraca.

Various processes, procedures, test equipment, and methods developed under this contract were reported in detail in the Final Report, June 17, 1968.

ABSTRACT

Stanford Research Institute, Menlo Park, California
DEVELOPMENT OF TECHNIQUES TO IMPROVE BLADDER MATERIALS AND TEST METHODS
Final Technical Report: Addendum One, March 30, 1969
T. F. Muraca, J. S. Whittick, and A. A. Koch
(JPL Contract No. 951484 under NAS7-100; SRI Project PRD-6068)

This Final Technical Report: Addendum One summarizes the work performed since the issuance of the Final Report (June 17, 1968) and does not repeat the details included in that report.

Equivalent PTFE films for expulsion bladders can be made from aqueous dispersions obtained from different suppliers without alteration of the process parameters developed previously (Final Report). The flex resistance of the PTFE films is not affected by prolonged immersion in dinitrogen tetroxide (N_2O_4).

The micro crease tester originally constructed at SRI was rebuilt from metals that are compatible with N_2O_4 as well as oxygen difluoride (OF_2), and the loading system was redesigned to provide smoother and more accurate performance at a constant tension.

The results of a comprehensive and critical literature review indicate the feasibility of cladding titanium alloys with an electroless nickel plate to protect them from impact sensitivity in the presence of OF_2 or possible hydrogen embrittlement in the presence of diborane (B_2H_6); a program of development for such a plate is outlined.

CONTENTS

ILLUSTRATIONS

I INTRODUCTION

This report is an Addendum to the Final Report published under JPL Contract No. 951484 on June 17, 1968. It summarizes the work performed by Stanford Research Institute in continuation of the program during the period August 1968 to March 1969.

The primary objectives of the program of work have been to investigate promising materials for expulsion devices and to develop methods for testing such materials.

During the prior contract period, process parameters were established for the fabrication of polytetrafluoroethylene films from aqueous dispersions that would be suitable for use in expulsion bladders, and procedures were developed for the physical, mechanical, and chemical characterization of the films.

Recent work has included a comparative study of PTFE films made from aqueous dispersions obtained from different sources in order to determine whether processing parameters would have to be modified or film properties would vary significantly. The results of this investigation are given in Section II.

The micro crease tester, originally designed and constructed for testing the crease-and-fold resistance of films in OF_2, has been rebuilt for testing films in the presence of N_2O_4. Essential features of design and construction are discussed in Section III, and the results of testing PTFE films in N_2O_4 are given in Section II.

The prior work also showed that available polymeric materials compatible with space storable propellants are unsuitable for use at the

semicryogenic temperatures required because of rapid embrittlement. Since titanium alloys are under serious consideration for the containment of storable propellants for long missions, it is possible they may be used for expulsion diaphragms also. However, titanium and its alloys are subject to pyrophoricity, impact sensitivity, hydrogen embrittlement, etc., in the presence of certain oxidizers and fuels. In order to ensure the successful use of these desirable alloys, it was suggested that the surfaces to be exposed could be protected by a thin coating of nickel applied by an electroless procedure. The feasibility of cladding the alloys with nickel and the attendant problems are discussed in Section IV; a program outline for developing and assessing a suitable electroless nickel plate for titanium and its alloys is given.

II A COMPARATIVE STUDY OF POLYTETRAFLUOROETHYLENE FILMS

In the work previously reported under this contract,* the aqueous dispersions of polytetrafluoroethylene used for the experimental fabrication of propulsion bladder films were obtained from a single source, E. I. du Pont de Nemours and Company. Since it is possible that similar dispersions obtained from different suppliers may require modified processing techniques or, more particularly, that the end products may have different properties, a one-gallon lot of PTFE aqueous dispersion was obtained from Du Pont ("Teflon" TFE-30) and also from Imperial Chemical Industries, Ltd. ("Fluon" GP-1). A film was prepared from each dispersion under identical conditions so that comparisons could be made of several properties of the resulting films.

Teflon TFE-30 dispersion contains 59-61% of PTFE solids with a particle size of 0.05 to 0.5μ and 5.5-6.5% of a nonionic wetting agent.

Fluon GP-1 dispersion contains 60% PTFE solids with a mean particle size of 0.2μ and 6% of a nonionic wetting agent.

Each dispersion was diluted so that the final mixture for spraying contained 30% of PTFE solids, and the wetting agent content was increased to 7% with Triton X-100. Sixteen spray applications of each mixture were used to build films of 5-mil thickness on the aluminum mandrels; each applied layer was sintered at 360°C for 20 minutes. Finally, the films were sintered at 360°C for 24 hours and then quenched by an instantaneous drop of the mandrel into a mixture of ice and water.

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* Final Report for June 17, 1968.

The only difference in physical appearance of the films is that the Fluon film is very nearly "water white" and shiny, whereas the Teflon film is slightly milky with a satin sheen.

The crystallinity of the two films was determined by the x-ray diffraction technique described in our earlier report and found to be:

Fluon GP-1	43.0% crystallinity
Teflon TFE-30	45.5% crystallinity.

Electronmicrographs of the films indicate structural differences at a magnification of 8,000 (Figures 1 and 2), but relatively no difference at a magnification of 100,000 as shown in Figures 3 and 4.

A series of crease-and-fold measurements was made for each film using the modified micro tester (described in Section III of this report) under a 2-lb tension with a blade angle of 2^{0} and a reference angle of 120^{0}. The differences in flex resistance are not considered to be significant.

Cycles-to-Failure (in Air)

	Fluon GP-1		Teflon TFE-30
	303		202
	304		252
	401		270
	387		264
	269		260
Av =	333	Av =	250

There has been some question as to whether the flex resistance of PTFE films as measured in air might be altered after the films were soaked in and tested under N_2O_4. Thus, a specimen of each film was soaked in N_2O_4 (liquid) for 20 hours and then flexed in the liquid. Every few cycles,

Figure 1 ELECTRONMICROGRAPH OF TEFLON TFE-30 FILM WITH 45.5% CRYSTALLINITY AT 8,000X; WO_3 SHADOWED, SiO REPLICA

Figure 2 ELECTRONMICROGRAPH OF FLUON GP-1 FILM WITH 43.0% CRYSTALLINITY AT 8,000X; WO_3 SHADOWED, SiO REPLICA

Figure 3 ELECTRONMICROGRAPH OF TEFLON TFE-30 FILM WITH 45.5% CRYSTALLINITY AT 100,000X; WO_3 SHADOWED, SiO REPLICA

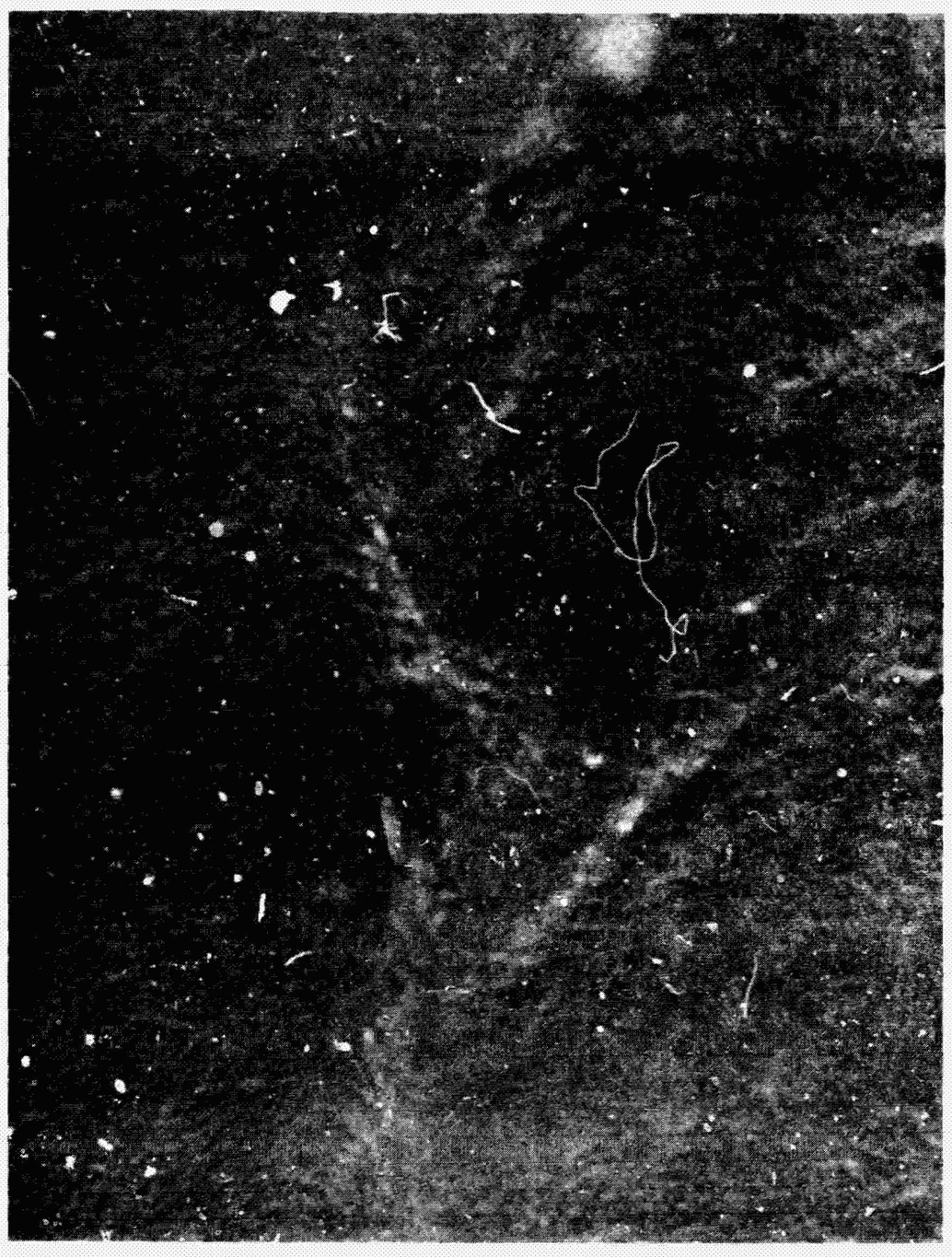

Figure 4 ELECTRONMICROGRAPH OF FLUON GP-1 FILM WITH 43.0% CRYSTALLINITY AT 100,000X; WO_3 SHADOWED, SiO REPLICA

the N_2O_4 was withdrawn and the system flushed with dry nitrogen prior to operating the spark hole-tester. The values obtained showed no significant difference from those given above:

Fluon GP-1	344 cycles-to-failure in N_2O_4
Teflon TFE-30	210 cycles-to-failure in N_2O_4

The results of the above experiments and tests indicate that propulsion bladders of equivalent quality can be made from PTFE aqueous dispersions furnished by different suppliers, that the originally developed process parameters need not be modified for different PTFE dispersions, and that the flex resistance of the PTFE films is not affected by contact of the films with N_2O_4 or its solution therein.

III MODIFICATION OF MICRO CREASE TESTER

The first micro crease tester constructed at SRI* was made of monel metal with various nickel or nickel-plated members since it was thought at the time that monel and nickel were the most suitable metals for use with OF_2. However, for the present work, flex measurements in the presence of N_2O_4 were required, and since nickel or nickel alloys are not compatible with N_2O_4, the apparatus was rebuilt entirely of aluminum alloys and stainless steel alloys which are compatible not only with N_2O_4 but also with OF_2. The spring-loaded system for applying tension to specimens being flexed was replaced with a gravity system (lead weights) to provide smoother, evener performance and a constant tension.

The inverted bell-jar (3"-diam Pyrex glass) used to house the first apparatus and contain the test medium was replaced by a 3"-diam Pyrex glass pipe, flanged at each end. This permits bottom loading of the sample under test, simplified positioning of the spark test probe, a drainage port for media that are fluid at room temperature, and an outlet for flushing the system with an inert gas to sweep out fumes of N_2O_4. This glass pipe housing is suitable for testing in propellants or other materials at room or at elevated temperatures. When materials are to be tested at less than room temperature, the bell-jar type of chamber is required since it can be immersed in a coolant bath.

Figure 5 is a close-up view of the apparatus showing a lead weight, a PTFE sample clamped in the test fixture around the crease fitting, and the flange system at either end of the Pyrex glass pipe. The valve at lower right leads from the liquid drain port to the exhaust system, the

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* Final Report for June 17, 1968 under this contract.

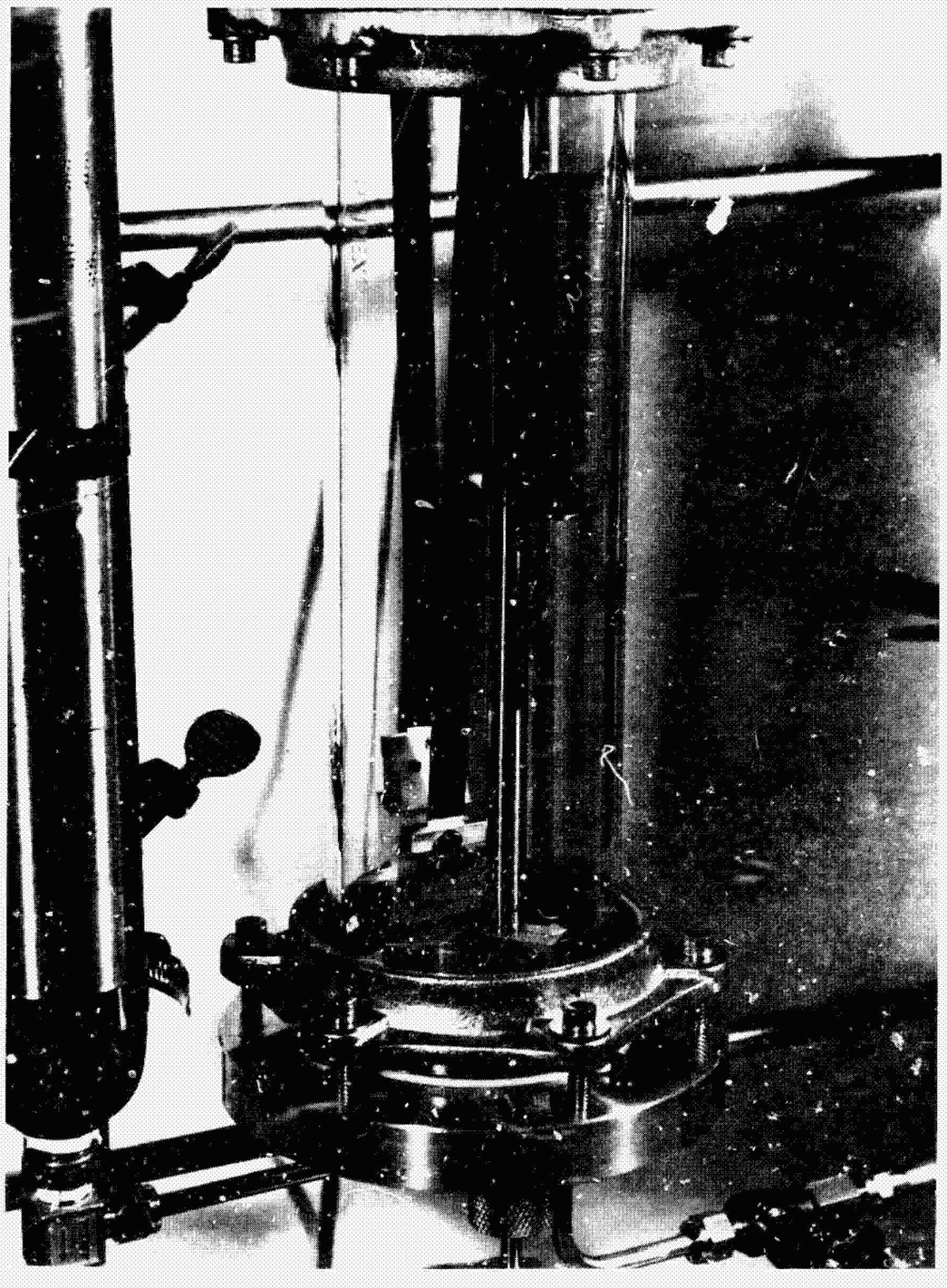

Figure 5 CLOSE-UP VIEW OF MODIFIED CREASE TESTER IN CLOSED CHAMBER

knurled knob at lower center is used to raise or lower the spark test probe as required, and the tubing shown at lower left leads to the gas exhaust system.

In Figure 6, the whole test assembly is seen, including the drive motor, with a cylinder of N_2O_4 in place; N_2O_4 is brought into the test chamber at upper left. (The glass monifold shown at the top of the photograph is not a part of this system.)

The over-all assembly drawing of the test apparatus is given in Figure 7, and the design drawing for the crease fitting in Figure 8. (Several complete sets of drawings, not included in this report, have been submitted to JPL.)

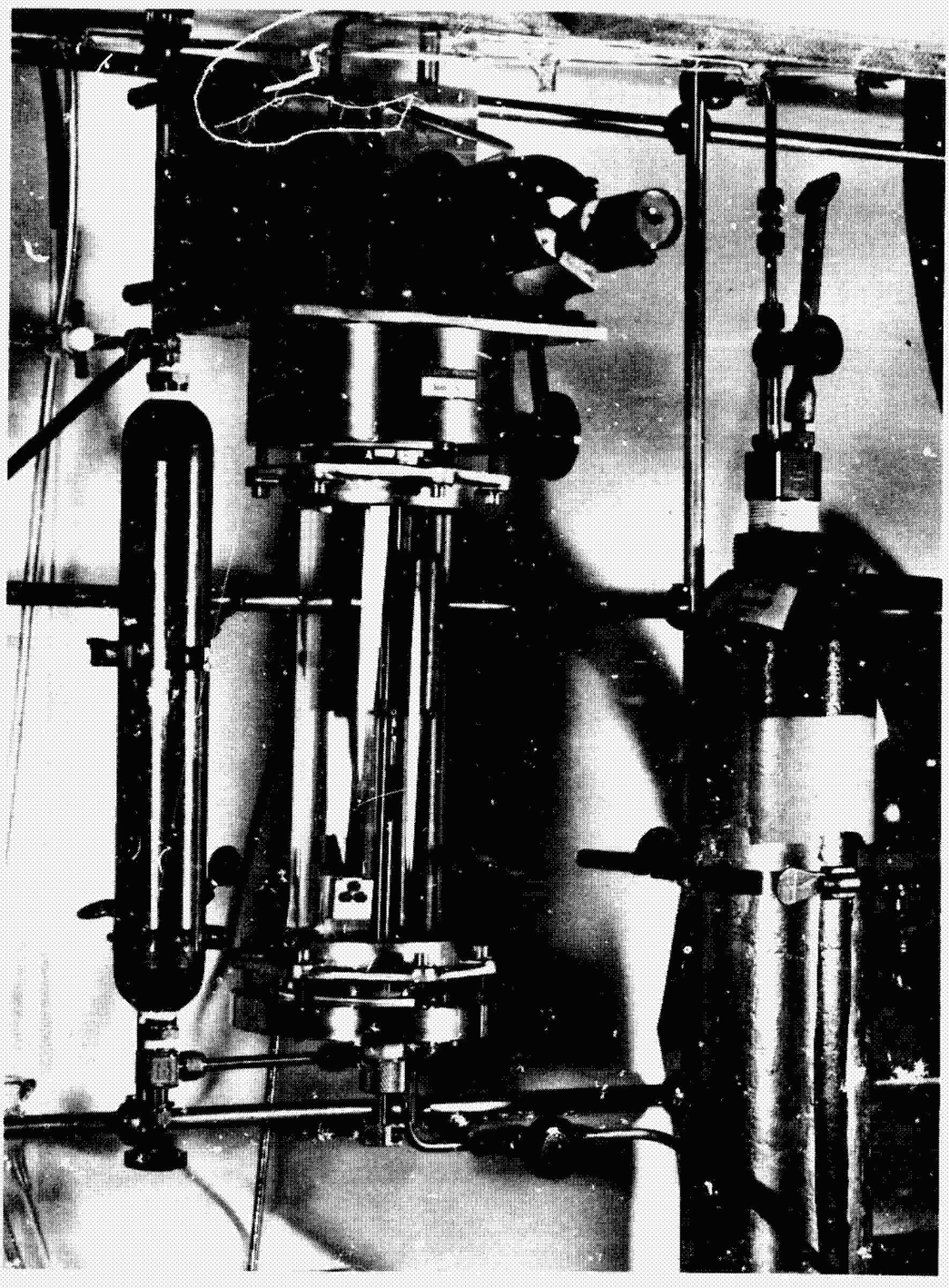

Figure 6 ASSEMBLY OF APPARATUS FOR CREASE TESTING

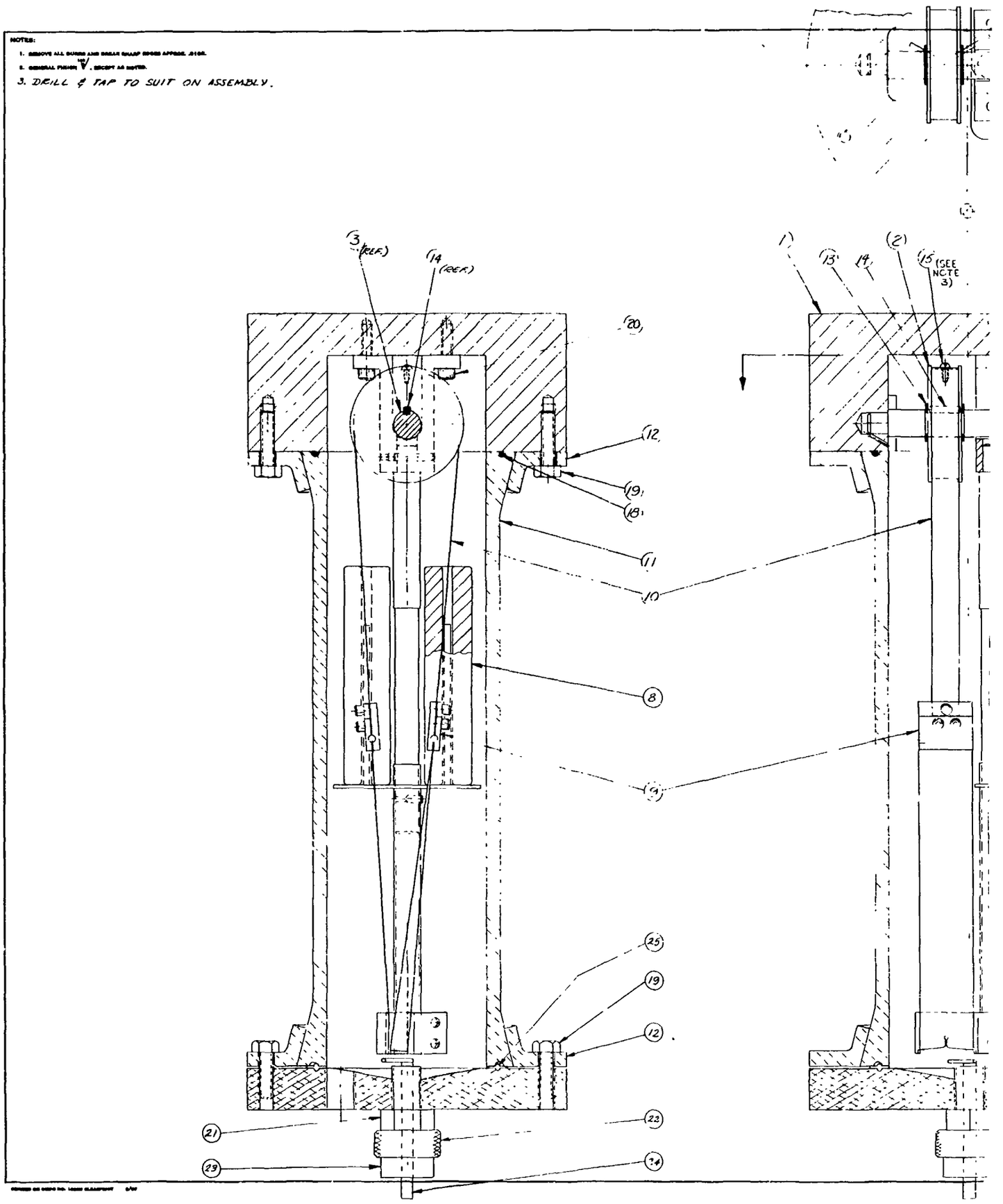

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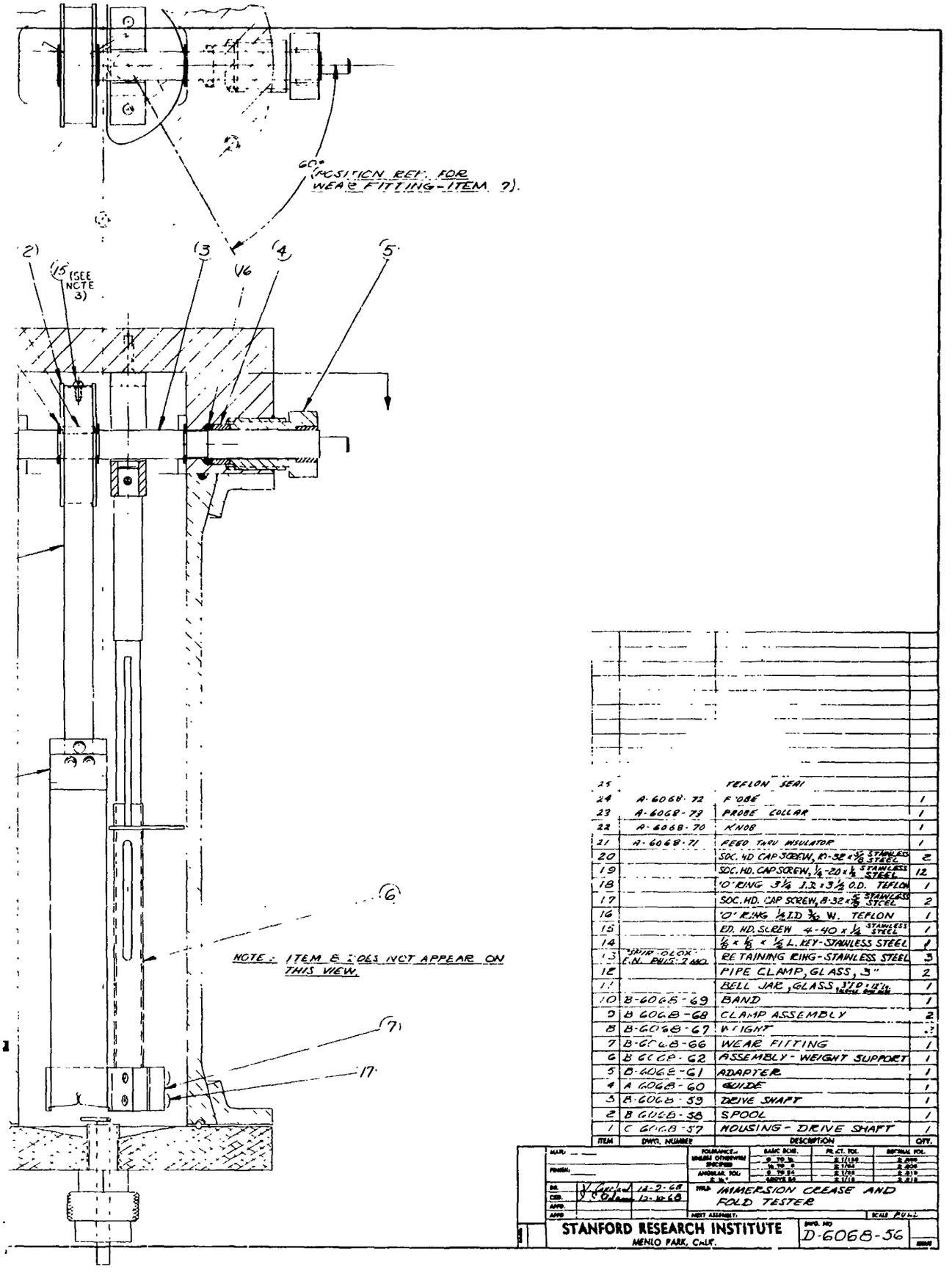
Figure 7 OVER-ALL ASSEMBLY DRAWING



ITEM	DWG. NUMBER	DESCRIPTION	QTY.
25		TEFLON SEAL	
24	A-6068-72	FOOT	1
23	A-6068-73	PROBE COLLAR	1
22	A-6068-70	KNOB	1
21	A-6068-71	FEED THRU INSULATOR	1
20		SOC. HD CAP SCREW, #0-32 x 5/16 STAINLESS STEEL	2
19		SOC. HD. CAP SCREW, 1/4-20 x 1/4 STAINLESS STEEL	12
18		'O' RING 3 1/4 I.D. x 3 1/2 O.D. TEFLON	1
17		SOC. HD. CAP SCREW, 8-32 x 5/8 STAINLESS STEEL	2
16		'O' RING 1/2 I.D. 3/16 W. TEFLON	1
15		ED. HD. SCREW 4-40 x 1/2 STAINLESS STEEL	1
14		1/8 x 1/8 x 1/2 L. KEY - STAINLESS STEEL	1
13	"SPIR-LOCK" E.N. PLUS 2 REQ.	RETAINING RING - STAINLESS STEEL	3
12		PIPE CLAMP, GLASS, 3"	2
11		BELL JAR, GLASS, 3.0 I.D. x 12 1/4 HEIGHT OUTSIDE	1
10	B-6068-69	BAND	1
9	B-6068-68	CLAMP ASSEMBLY	2
8	B-6068-67	WEIGHT	1
7	B-6068-66	WEAR FITTING	1
6	B-6068-62	ASSEMBLY - WEIGHT SUPPORT	1
5	B-6068-61	ADAPTER	1
4	A-6068-60	GUIDE	1
3	B-6068-59	DRIVE SHAFT	1
2	B-6068-58	SPOOL	1
1	C-6068-57	HOUSING - DRIVE SHAFT	1

MAT'L: —

FINISH: —

DR.	V. Cuglietta	12-2-68
CHK.	[illegible]	12-10-68
APPD.		
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TOLERANCE — UNLESS OTHERWISE SPECIFIED

BASIC DIM.	FR. CT. TOL.	DECIMAL TOL.
0 TO ½	± 1/64	± .020
½ TO 6	± 1/32	± .005
6 TO 24	± 1/16	± .010
OVER 24	± 1/8	± .020

ANGULAR TOL. ± ½°

TITLE: IMMERSION CREASE AND FOLD TESTER

NEXT ASSEMBLY: —

SCALE: FULL

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MENLO PARK, CALIF.

DWG. NO. D-6068-56

LY DRAWING OF CREASE TESTER

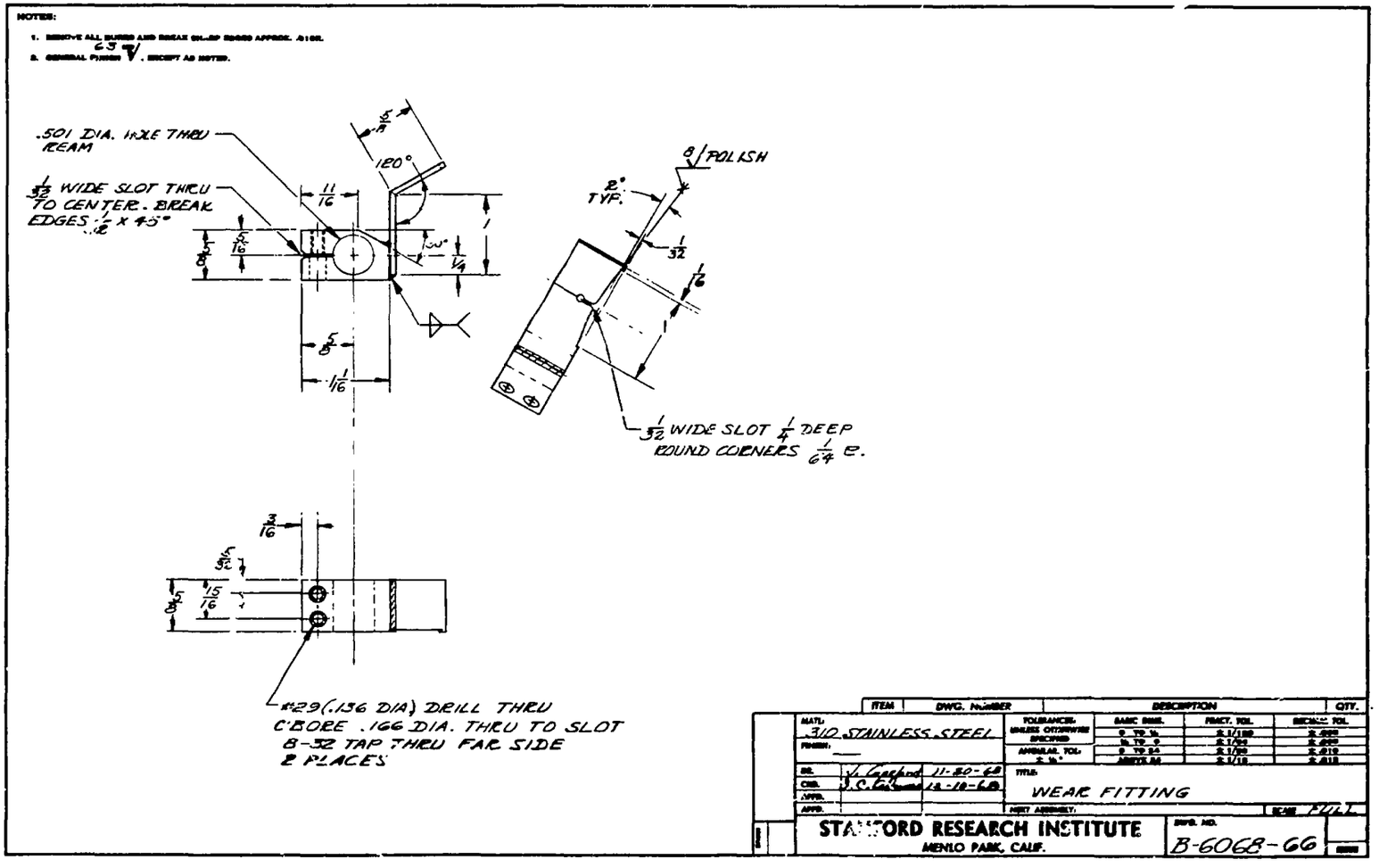

Figure 8 DESIGN DRAWING OF WEAR FITTING

IV NICKEL CLADDING OF TITANIUM ALLOYS

The potential value of titanium and its alloys as aerospace materials led to the accelerated development of processing and fabrication techniques, sponsored by several Government agencies since World War II. The high strength-to-weight ratio and resistance to atmospheric corrosion were but two of the attractive properties, and the development of alloys progressed toward further improvement of mechanical properties; thus, the alloy currently most favored for space use, 6Al-4V, was introduced in 1954.

During the early years of titanium alloy development, the quality of performance was proven in uses as structural members and components for aircraft, rockets, weapons, process equipment, etc. Titanium and its alloys have been used successfully for pressure vessels in spacecraft (Gemini and Ranger) and for storage of fuels such as hydrazine (Surveyor and Mariner). The mechanical properties of 6Al-4V titanium alloy make it especially desirable for the containment of space-storable propellants, which are required for longer-duration space missions. However, there are uncertainties about its behavior in direct contact with at least two of the propellants under consideration, OF_2 and B_2H_6.

A recent survey [18] indicated that titanium alloy apparently has a satisfactory resistance to corrosion by OF_2, but has tendencies to impact sensitivity when in contact with it. There is also the possibility of hydrogen embrittlement and subsequent weakening of titanium alloys in the presence of B_2H_6, which undergoes thermal decomposition readily, but no information was found to clarify this point. In order to circumvent the possibility of hazards or malfunctions connected with impact sensitivity or hydrogen embrittlement, it was recommended [18] that a program be

established to investigate the plating of titanium alloys with a thin coating of nickel, which is resistant to corrosive attack by OF_2 or B_2H_6, is sufficiently ductile to withstand without crazing the motions involved in diaphragms, bellows, or valve movements, and is insensitive to impact in the presence of reactive propellants, thus protecting the titanium alloy system components. It was recommended further that electroless nickel procedures particularly be investigated in order to ensure the cladding and protection of even the generally inaccessible inner areas of transfer tubing and valve bodies, the thin edges or sharp creases of bellows, etc.

Another reason for cladding titanium or its alloys is to remove some undesirable properties of the bare metal that are displayed in the performance of moving parts; for example, a severe tendency to galling and low wear resistance. In fact, it is especially for this reason that electrolytic and electroless procedures have been investigated from time to time for cladding titanium and its alloys with nickel.

Before establishing an experimental program of work, available literature was reviewed to affirm the choice of an electroless nickel-plating procedure, to determine which parameters of electroless procedures had been studied definitively, particularly with application to titanium alloys, and to identify problem areas. The complete bibliography is given at the end of this Section.

A. Discussion

It is conceded generally that titanium can be plated with nickel by standard electrodeposition methods, but the plate is loosely adherent, apparently due to an initial surface film of TiO_2 which is formed readily on a "clean" titanium surface in air or in aqueous solutions. The procedures of various workers for removing this surface film of TiO_2 prior to electroplating have included anodic etching, chemical etching, wet and dry sandblasting, and fused salt pickling; however, reported good results have

not been consistently reproducible by other workers. Attempts at diffusion annealing after plating have resulted in more tightly adherent coatings, but with a general loss in surface mechanical properties.

Since the discovery of an electroless procedure for nickel plating in 1944, commercial use has been increasing steadily because of its many advantages over electrodeposition methods such as: uniformity of deposit; lesser porosity of deposit; good coverage of inner areas, crevices, protrusions, etc., without need for complex configurations of anodes; and fewer problems with hydrogen embrittlement. One limitation is that electroless nickel may be deposited only at a rate of less than one mil per hour in contrast with the higher rates afforded by electrodeposition. Coatings over 5 mils in thickness are not economically practical because of the costs of the reducing agents, but it is estimated that no more than about 1-mil thickness will be required for the protection of titanium alloys for the intended purposes.

The most widely used electroless procedure utilizes an acid bath (pH 4-6) containing a nickel salt and a hypophosphite reductant, with additives for bath quality or plate finish; the reduction of the salt to nickel is catalyzed at and by surface of the metal being plated. (Less used is an alkaline bath, pH 8-10.) The plate is somewhat brittle, but made more ductile by annealing at about 400°C; it has a hardness and wear resistance greater than electroplated nickel and a greater resistance to chemical corrosion. However, it is an alloy of phosphorus and nickel containing from 3% to 15% phosphorus; the resistance of this alloy to corrosive attack by OF_2 is not known, nor is it known whether the presence of the phosphorus would enhance the impact sensitivity of the "nickel" plate in the presence of OF_2.

Experimental work has been performed with other reductants such as borohydrides and aminoborane; in these instances, a nickel alloy plate

with a boron content of 3% to 10% has shown excellent wear and corrosion resistance. On the other hand, with the use of hydrazine as a reductant, plates of 97-99% nickel were achieved, but 8-10 elements were present as impurities; there was a high gas content of 0.3-0.8% (oxygen, nitrogen, and hydrogen); and voids were created by decomposition of impurities during heating. Again, the reactivity with OF_2 of any of the above plates has not been investigated.

The same problem of surface treatment to remove the film of TiO_2 exists, of course, as in electrodeposition. There have been a number of attempts, recommendations, and patents for activating the surface, which must be examined critically not only from the point of view of promoting the adherence of the plate but also for the possible effect of impurities introduced from this source on the corrosion resistance and the impact sensitivity of the plate.

Final annealing of the plate is a critical factor in promoting and maintaining good physical and mechanical properties. Annealing has been performed under a variety of conditions, and some success has been achieved in producing electroless nickel plates on titanium alloys with mechanical properties approaching those of electrodeposited nickel on nonrefractory metals.

B. Future Work

In view of the above findings, it appears that the program of development required to ensure a protective coating for titanium and its alloys in the presence of advanced propellants should include:

(1) The selection or development of a reproducible and reliable surface treatment of the metal prior to plating;

(2) The selection or development of electroless plating baths and procedures for their maintenance for efficient and reproducible platings;

(3) The determination of the optimum annealing conditions.

Other determinations to be made in line with and to assess the course of the work include:

(1) The compositional analysis of plating baths at specific times and the relationship to the compositional analysis of the final plates;

(2) Photomicrographic inspection of plates for integrity and structural characteristics;

(3) Resistance of plates to bending or flexing;

(4) Resistance to impact sensitivity in the presence of advanced propellants.

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